Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N'-[Bis(methylthio)methylene]cyanoacetohydrazide

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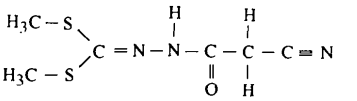
(*Received 17 January 1996; accepted 30 May 1996*)

Abstract

The title compound, $C_6H_9N_3OS_2$, forms chains of hydrogen-bonded molecules along the [100] direction, the chains being held together by van der Waals interactions. Bond lengths and angles are in good agreement with standard literature values.

Comment

In the course of a project aimed at finding possible new products, attention has been focused on the use of cyanoacetohydrazide derivatives as drugs (Negwer, 1987). In this context, the title compound, (I), has been synthesized and characterized.



(I)

The title compound was obtained from cyanoacetohydrazide by adding carbon disulfide and methyl iodide at room temperature (Napoles, Peseke & Quincoces, 1987), and displays biological activity against fungus and parasites of bovine cattle (Napoles, 1993). The product of the above reaction was characterized by quantitative analysis, IR and proton-NMR spectroscopy, and mass spectrometry (Napoles, 1993), and its crystal structure was determined by single-crystal X-ray diffraction (Fig. 1).

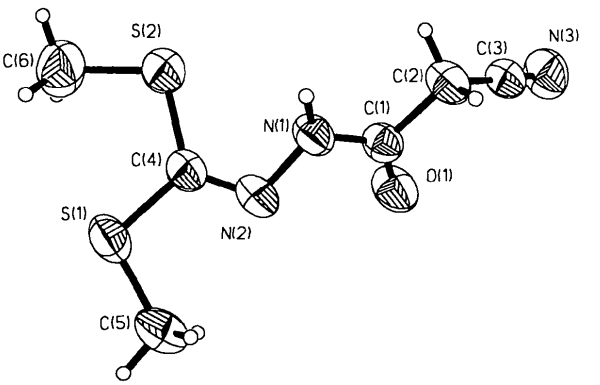


Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids.

Bond lengths and angles in (I) are all in good agreement with literature values. In particular, those of the cyanoacetohydrazide moiety are very similar to the corresponding values found in α-cyanoacetohydrazide itself (Chieh, 1973). The only significant difference is in the C(2)—C(1)—N(1)—N(2) torsion angle [165.9 (5) in the title compound and 172.6° in α-cyanoacetohydrazide]. This is probably as a result of the substituent on the N(2) atom causing the molecule to deviate from planarity; the dihedral angle between the planes of the $(MeS)_2CN$ and $HNC(=O)CH_2CN$ moieties is 55.8 (5)°.

The crystal packing includes infinite chains of hydrogen-bonded molecules along the [100] direction (Fig. 2). The amine N(1) atom of each molecule is hydrogen bonded to the carbonyl O(1) atom of the molecule of the next unit cell (Table 2). The chains are held together in the crystal by means of van der Waals interactions.

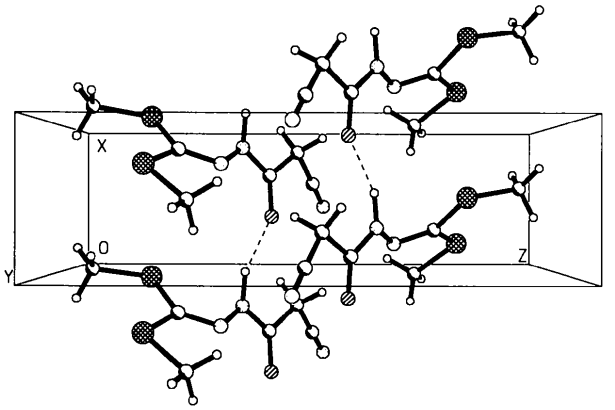


Fig. 2. Partial packing diagram viewed along the *b* axis, showing the hydrogen bonding in two adjacent chains.

Experimental

The title compound was obtained from cyanoacetohydrazide by the addition of carbon disulfide and methyl iodide at room temperature (Napoles, Peseke & Quincoces, 1987). Crystals suitable for X-ray analysis were obtained by evaporation from ethanol solution.

Crystal data

$C_6H_9N_3OS_2$
M_r = 203.28
Orthorhombic
$P2_12_12_1$
a = 4.634 (1) Å
b = 13.069 (4) Å
c = 15.832 (5) Å
V = 958.8 (5) Å^3
Z = 4
D_x = 1.41 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 25 reflections
θ = 14–18°
μ = 4.72 mm^{-1}
T = 293 K
Prism
0.62 × 0.20 × 0.14 mm
White

Data collection

Nicolet $R3$ diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.33, T_{max} = 0.54
737 measured reflections
737 independent reflections
680 observed reflections [$F > 4\sigma(F)$]

θ_{max} = 55°
$h = 0 \rightarrow 4$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 16$
2 standard reflections monitored every 50 reflections
intensity decay: 5%

Refinement

Refinement on F
R = 0.039
wR = 0.050
S = 1.13
680 reflections
113 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0012F^2]$

$(\Delta/\sigma)_{max}$ = 0.077
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S(1)	0.2109 (4)	0.5561 (1)	0.7877 (1)	0.068 (1)
S(2)	0.5141 (4)	0.7593 (1)	0.7911 (1)	0.073 (1)
O(1)	−0.1072 (8)	0.8186 (3)	0.5709 (3)	0.072 (2)
N(1)	0.3261 (10)	0.7759 (3)	0.6240 (3)	0.054 (1)
N(2)	0.2199 (10)	0.6846 (3)	0.6600 (2)	0.055 (1)
N(3)	−0.0474 (13)	1.0518 (4)	0.4719 (3)	0.077 (2)
C(1)	0.1528 (11)	0.8302 (3)	0.5753 (3)	0.049 (2)
C(2)	0.3073 (12)	0.9125 (4)	0.5226 (3)	0.059 (2)
C(3)	0.1046 (12)	0.9900 (4)	0.4932 (3)	0.056 (2)
C(4)	0.3061 (11)	0.6700 (3)	0.7361 (3)	0.053 (1)
C(5)	−0.0010 (5)	0.4937 (4)	0.7089 (4)	0.077 (2)
C(6)	0.571 (2)	0.7031 (5)	0.8928 (4)	0.097 (3)

Table 2. *Selected geometric and hydrogen-bonding parameters (Å, °)*

S(1)—C(4)	1.755 (5)	S(1)—C(5)	1.785 (6)
S(2)—C(4)	1.746 (5)	S(2)—C(6)	1.789 (6)
O(1)—C(1)	1.216 (6)	N(1)—N(2)	1.411 (6)
N(1)—C(1)	1.320 (7)	N(2)—C(4)	1.284 (6)
N(3)—C(3)	1.124 (7)	C(1)—C(2)	1.538 (7)
C(2)—C(3)	1.458 (7)		
C(4)—S(1)—C(5)	101.6 (2)	C(4)—S(2)—C(6)	104.8 (3)
N(2)—N(1)—C(1)	118.6 (4)	N(1)—N(2)—C(4)	113.4 (4)
O(1)—C(1)—N(1)	124.7 (5)	O(1)—C(1)—C(2)	121.1 (5)
N(1)—C(1)—C(2)	114.2 (4)	C(1)—C(2)—C(3)	111.0 (4)
N(3)—C(3)—C(2)	178.0 (6)	S(1)—C(4)—S(2)	118.3 (3)
S(1)—C(4)—N(2)	119.0 (4)	S(2)—C(4)—N(2)	122.7 (4)

D—H···A	D—H	H···A	D···A	D—H···A
N(1)—H(1)···O(1')	1.00 (7)	1.86 (6)	2.813 (6)	157 (6)

Symmetry code: (i) $1 + x, y, z$.

H atoms were refined with a riding model and fixed U_{iso} values (0.08 Å^2), except for the H(1) atom bonded to N(1), for which the position was freely refined with a U_{iso} value of 0.06 Å^2.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*.

The authors thank The Third World Academy of Science for financial support through TWAS research grant 94-001 RG/CHE/LA.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1085). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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